

LABNOTES

Spring 1997



The Newsletter of the Wisconsin Laboratory Certification and Registration Program
Program Info: (608) 267-7633 Program Fax: (608) 266-5226

Report: Retain Certification Standards Review Council

Over the past year, Wisconsin Lieutenant Governor Scott McCallum has been reviewing the functions of 144 statutory state councils, boards and commissions. His final report, issued February 21, 1997, recommends retaining only 63 of these groups and dissolving the remainder. The Certification Standards Review Council was listed among those that should be retained. This council is a nine member advisory body to the Wisconsin Department of Natural Resources Laboratory Certification Program. Its members represent a diverse group of environmental interests throughout the state including municipal, industrial and commercial laboratories. The Lt. Governor's report also recommends changing the statutory requirement that one member of the council be a "farmer actively engaged in livestock production". This position has been extremely difficult to fill in the past and is currently vacant. The report suggests that the statute should allow greater flexibility in choosing a representative from the agricultural industry who is interested in data quality issues.

The statutory functions of the council are not expected to change as a result of the report. Currently, the statute authorizes the council to provide input to the department on work planning, fees, budget issues and variance requests. The council also makes recommendations to the department concerning the specification of test categories, reference samples, standards for certification

and other administrative aspects of the program. See page 3 of this issue of *LabNotes* for a list of current council representatives.

It's renewal time again!

Once again, it is time to start thinking about renewing your laboratory's certification or registration for fiscal year 1998 (July 1, 1997 - June 30, 1998). Check your certificate for the certification ending date. If your laboratory's certification or registration will expire June 30, 1997, please make sure that you have completed all of the requirements for renewal. To avoid delays in receiving your new certificate, your laboratory must:

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(Renewal, from page 1)

- ◆ Submit acceptable reference sample results for each test or test category prior to the June 30 deadline. Consult the program information booklet or the web site for a list of approved providers.
- ◆ Pay the annual fee prior to the June 30, 1997 deadline. You will receive your certification bill in May. This bill will coincide with any other Wisconsin environmental fees that your facility is required to pay (e.g., NR 101).

Your laboratory will not be renewed until we receive acceptable reference sample results and full payment of fees. If your laboratory analyzes reference sample from a private provider or the New York Department of Health, it is your responsibility to forward these results to the Wisconsin DNR (we do not automatically receive these results). Your laboratory will be assessed a late fee if we do not receive your payment within thirty days of the due date. After September 1, the department will not renew your laboratory's certification or registration and you will have to submit a new application for certification to be reinstated.



LabNotes - Newsletter of the Laboratory Certification Program

LabNotes is published twice annually by the Wisconsin DNR Laboratory Certification and Registration Program. For information about distribution or to make suggestions for future articles, contact the editor at (608) 267-0579.

George E. Meyer,
Secretary, Wisconsin Dept. of Natural Resources

Jim Addis,
Director, Bureau of Integrated Science Services
(608) 266-0837

John R. Sullivan,
Chief, Analytical and Statistical Services
(608) 267-9753

Jeffrey Ripp,
Editor, LabNotes Newsletter
(608) 267-0579

Reminder to Review Your Certificate!

Once you have paid your fees and passed your reference samples, the DNR will send your laboratory a new certificate. It is very important that you check your certificate carefully for errors. If the certificate does not reflect your expectations, please contact Mike Kvitrud at (608) 261-8459 or by e-mail: mkvitrud@dnr.state.wi.us as soon as possible to resolve the problem.

A short guide to abbreviations and acronyms used in LabNotes

The environmental field is fraught with jargon and acronyms. Unfortunately, it is difficult to convey current issues and events in *LabNotes* without reverting to the acronyms. Refer to this list if you are struggling with your "ABC's".

AA-	Atomic Absorption
APG-	Analytical Products Group, Inc.
ASI-	Analytical Standards, Inc.
CWA-	Clean Water Act
DMR -	Discharge Monitoring Report
DNR -	Wisconsin Department of Natural Resources
DRO -	Diesel Range Organics
ECD -	Electron Capture Detector
ELCD -	Electrolytic Conductivity Detector
EPA -	Environmental Protection Agency
ERA -	Environmental Resource Associates
FID -	Flame Ionization Detector or Facility Identification Number
GC -	Gas Chromatography
GC/MS-	Gas Chromatography-Mass Spectrometry
GRO -	Gasoline Range Organics
HPLC -	High Performance Liquid Chromatography
LOD -	Limit of Detection
LOQ -	Limit of Quantitation
MDL -	Method Detection Limit
MS-	Mass Spectrometry
NPD -	Nitrogen Phosphorus Detector
NYDOH-	New York Department of Health
OSHA -	Occupational Safety and Health Act
PCB -	Polychlorinated Biphenyl
PID -	Photo Ionization Detector
PVOC-	Petroleum Volatile Organic Compound
SLH -	State Laboratory of Hygiene
VOC -	Volatile Organic Compound

1997 Certification Standards Review Council Representatives

Commercial Laboratory

Ms. Mary Christie, Chair
En Chem, Inc.
205 Seagull Drive
Mosinee, WI 54455
(715) 693-1953

Public Water Utility

Ms. Ruth Klee Marx
County of Marathon Health Dept.
1200 Lake View Drive, Room 200
Wausau, WI 54403-6797
(715) 842-7891 ext. 337

State Laboratory of Hygiene

Dr. Bill Sonzogni
State Lab of Hygiene
465 Henry Mall
Madison, WI 53706
(608) 262-8062

Large Municipal Wastewater Plant

Mr. John Moser
Milwaukee Metro. Sewerage Dist.
250 w. Seeboth Street
Milwaukee, WI 53204-1446
(414) 277-6378

Small Municipal Wastewater Plant

Mr. Gilbert Williams, Vice Chair
Sun Prairie Water Pollution Control Facility
300 East Main
Sun Prairie, WI 53590
(608) 837-6292

Industrial Laboratory

Mr. David Kollakowsky
Wisconsin Electric Power Co.
PO Box 2046
Milwaukee, WI 53201-2046
(414) 221-2835

Solid and Hazardous Waste Disposal Facility

Ms. Barbara Hill
WMX Env. Monitoring Labs
2100 Cleanwater Drive
Geneva, IL 60134
(630) 208-3100 ext. 112

Interest in Laboratory Certification

Mr. Russell Janeshek
Foth & Van Dyke
PO Box 19012
Green Bay, WI 54307-9012
(414) 497-2500

Natural Resources Board approves fees for 1998 fiscal year

The Natural Resources Board approved the proposed laboratory certification and registration fees for fiscal year 1998 at its March 26, 1997 meeting. The fees are adjusted each year according to the formula in Chapter NR 149, Wis. Adm. Code. The formula is based upon the legisla-

tively approved spending authority and the number of laboratories participating in the program. While the program's expenses haven't increased significantly, the fees approved by the Board represent a slight increase over last year due to a drop in the number of certified and registered laboratories. The cost per relative value unit will increase from \$35 to \$37.50, which represents about a \$125 increase for a typical commercial laboratory and a \$33 increase for a typical wastewater treatment plant laboratory. The fees are anticipated to generate revenue equal to the amount collected in fiscal year 1997. Due to the fee increase, any application forms that you may have at your laboratory will be outdated. Please contact John Condron at (608) 267-2300 to get a copy of the most recent application form.

1998 Certification & Registration Fees	
Program Base Fee:	\$375.00
Cats. 01 - 04:	\$37.50
Cats. 07 - 14, 16 & 19:	\$75.00
Cats. 15 & 17:	\$450.00
Cat. 18:	\$750.00
Cat. 20:	\$975.00
Minimum & Reciprocity Fee:	\$900.00
Initial Application Fee:	\$225.00
Revised Application Fee:	\$125.50
Late Payment Fee:	\$75.00

Update: The future of the EPA reference sample programs

Laboratories using the Discharge Monitoring Report - Quality Assurance (DMR-QA) reference samples for Wisconsin certification or registration received a letter from the DNR last December. The letters were mailed to all laboratories whose certification or registration period expires on December 31 each year. In the letter, the DNR suggested that these laboratories consider using another reference sample provider for next year's renewal, beginning January 1, 1998. Due to the comments received both by phone and during the recent public meetings, the Laboratory Certification Program has decided to adjust its renewal schedule around the DMR-QA samples for one more year. This means that for next year, your laboratory can still use the DMR-QA samples for certification or registration. If the DMR-QA results are late, as they historically have been, we will once again extend the renewal deadline until the results are available. The program still advises that laboratories use another provider, but will be flexible for those labs that are federally required to analyze the DMR-QA's. Permittees should have already received a DMR-QA Study #17 announcement booklet. If a permittee has not yet received this or does not understand their requirements, please contact Michael Kvitrud at (608) 261-8459 or by e-mail at mkvitrud@dnr.state.wi.us.

On a related note, EPA is considering several options for externalizing their reference sample programs, including dropping the programs entirely at the end of the federal fiscal year (Oct. 1, 1997). This means that the current EPA Water Supply (WS039), Water Pollution (WP038) and DMR-QA 17 may be the last round of samples offered. A briefing of EPA senior management was conducted on February 18, 1997 and more discussions regarding externalization of the programs were held in

March. A decision regarding the samples has not been made, but we will continue to keep our laboratories informed as we become aware of new information. In the meantime, we suggest that all of our non-safe drinking water laboratories look into receiving their reference samples from an alternate provider. **Safe drinking water labs are still required to analyze the EPA's Water Supply (either WS 038 or 039) samples for renewal.** In the event that the EPA studies are no longer offered after October 1, 1997 the Wisconsin DNR will approve the use of an alternate provider. Watch for more information about reference samples in the fall issue of *LabNotes* or on the lab certification web site at:

<http://www.dnr.state.wi.us/eq/lc/>.

Reminder #1:

The EPA has now assigned lab codes to in-house (registered) labs. This means that permittees may have both a National Pollutant Discharge Elimination System permit number ("WI" followed by 7 digits) for the facility and a lab code ("WI" followed by 5 digits) for the in-house lab. The lab code should be used on the EPA's ordering form. The form for reporting results should contain both the discharger's permit number and the lab codes of any laboratories (including the in-house lab) which analyzed the samples.

Reminder #2:

The DMR-QA Study #17 is being combined with EPA's Water Pollution Study WP038. This means a commercial lab only has to order and analyze the WP038 samples and can report those results on the DMR-QA 17 reporting forms for their clients.

Introducing Wisconsin's two new audit chemists

A SHORT BIOGRAPHY BY GREG PILS...

It all started in a Marathon County, Wisconsin hospital in 1969. In the early hours of that cold February morning, a child was born. He was given the name Greg Pils, and 28 years later the fates would deliver him to the Wisconsin Department of Natural Resources. In this most hallowed of theaters, he was assigned a new role on life's great stage: that of the Laboratory Certification Program's most recent addition to their staff of audit chemists.

That new audit chemist is me. I received a Bachelor of Science degree in chemistry from the University of Wisconsin- Madison in December 1991, and since 1992 I have worked as an analyst in the Wisconsin environmental laboratory community. My primary areas of expertise are volatile organics, diesel range organics and gasoline range organics, flame and graphite furnace atomic absorption, and cold-vapor mercury analysis.

One of the goals I have set for myself in my new position is to bring the perspective of the private laboratory to discussions of departmental policy and philosophy. Any input you may have that would aid me in the realization of this goal is welcome. I can be reached by phone at (608) 267-9564, or by e-mail at pilsg@dnr.state.wi.us.

I'll doubtlessly be meeting several of you in the coming months, and I look forward to establishing many productive, mutually rewarding relationships as we close out the '90s and move forward into the next millennium.

A SHORT BIOGRAPHY BY DIANE DRINKMAN...

After a 4½-year stint at the University of Wisconsin-Madison Safety Department as a chemical management specialist, I decided to return to the realms of environmental chemistry as an audit chemist with the Laboratory Certification Program. In my years with the University, I acquired experience with compliance issues related to many federal and state regulatory programs. I also managed the campus Storm Water Discharge Permit Program, co-authored the *Chemical Safety and Disposal Guide* (© 1993, University of Wisconsin Board of Regents), presented papers at national, regional and local environmental health and safety conferences and worked with other state agencies on liability issues, the state hazardous waste disposal contract and numerous other regulatory matters.

Prior to working at the University of Wisconsin, I worked as an environmental chemist with Warzyn in Madison and at Badger Army Ammunition Plant, Olin Corporation, Baraboo. I have extensive experience with a variety of methods including, but not limited to volatile organics, polychlorinated biphenyls, nitroaromatics, metals, gas chromatography-mass spectrometry, other inorganics and the toxicity characteristic leaching procedure.

I feel that my unique combination of bench chemistry, regulatory compliance, education and information sharing will help me to serve the laboratory community. I am looking forward to meeting and helping each and every one of you, as best that I can. Please feel free to contact me at (608) 264-8950 or by e-mail at drinkd@dnr.state.wi.us if you have questions or concerns.



Alternate catalysts available for total kjeldahl nitrogen

Mercury, in the form of mercuric sulfate, is the catalyst required for the determination of total kjeldahl nitrogen (TKN). However, minimizing the sources of mercury contamination is a worthwhile goal, especially since the department is interested limiting discharges to the environment and improving laboratory detection limits. Several alternatives to mercury exist. In many matrices, selenium has been demonstrated to be as effective as mercury. Unfortunately, selenium is also a very toxic metal. *Standard Methods for the Examination of Water and Wastewater* (19th edition) suggests using a copper catalyst, with less sulfuric acid in the digestion reagent. Method 4500-N_{org} B of the 19th edition explains the specific requirements for using the copper catalyst (page 4-92, sec. 4).

To substitute other catalysts in place of mercury, a laboratory must demonstrate complete digestion using at least four replicates of 0.300 g of reagent grade nicotinic acid. Recoveries between 95 to 105% are required to allow the substitution. Nicotinic acid, also known as niacin, is 3-pyridinecarboxylic acid. Its molecular weight and nitrogen content are 123.11 and 11.38%, respectively. Questions about alternate catalysts should be addressed to Alfredo Sotomayor, (608) 266-9257 or by e-mail at sotoma@dnr.state.wi.us.

DNR recommends graphite furnace for molybdenum in wastewater sludge

The Fall 1996 issue of *LabNotes* identified molybdenum as a pollutant of concern in wastewater treatment sludge because of the low ceiling concentration requirement (75 mg/kg on a dry weight basis) for land-

spreading in chapter NR 204, Wis. Adm. Code. Molybdenum is often present in sludge because molybdenum salts can replace chromium in chemical treatments for controlling biological growth in cooling towers. Based on initial test results, it appeared that a number of wastewater facilities' sludge exceeded the molybdenum limit. Upon further investigation, department staff discovered that many of these apparent exceedances could be attributed in part to poor sensitivity of the flame atomic absorption method. This is exacerbated when low level detects are converted to mg/kg and then adjusted for the moisture content of the sample. Although the flame atomic absorption method is still approved in NR 219, Wis. Adm. Code, the department is discouraging laboratories from using this method to determine molybdenum in sludge. Instead, the DNR is recommending that laboratories confirm any molybdenum results above 75 mg/kg dry weight with a graphite furnace atomic absorption method before releasing the results to their clients.

Low level mercury: DNR moves towards pollution prevention

The DNR's Wastewater Program began looking at low level mercury data for influents and effluents about seven years ago. Since then, municipal facilities have faced permit limits as low as 2 ng/L but have been forced to use analytical methods with inappropriate detection limits. Several facilities repeatedly detected mercury in their effluent, sometimes above the quantitation limit. These results proved to be false positives attributed to contamination introduced in both the sampling and analytical processes. Actual mercury levels were found to be as much as two orders of magnitude

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lower than initially determined. Through this process, the department has gained valuable insight about the quality of mercury monitoring data and has concluded that continuing to monitor for mercury as we have in the past is a waste of time and money.

The May 1996 *Wisconsin Strategy for Regulating Mercury in Wastewater* is a result of the DNR's investigation of the available options for regulating mercury and their effectiveness. The strategy relies on pollution prevention rather than regulating mercury at part-per-trillion levels in treatment plant effluents. Implementing this strategy involves identifying sources contributing mercury to the plant, reducing the quantities discharged (pollution prevention) and tracking the effectiveness of the effort by monitoring mercury levels in treatment plant influents.

A key factor in the success of this strategy will be to overcome the limitations of existing analytical techniques. The EPA recently published methods that can realistically meet the analytical objectives. Certified laboratories that want to take advantage of these methods and provide low level mercury testing services can be recognized through the emerging technology provision of NR 149, Wis. Adm. Code. To meet the criteria for certification, a laboratory must demonstrate that: (1) the method chosen provides a sensitivity of 20 to 50 ng/L or lower in a wastewater matrix, (2) ambient contamination in the laboratory has been controlled, and (3) it can meet the specified quality criteria. The DNR will maintain a list of certified laboratories that successfully complete the approval process. To date only one laboratory, Northern Lake Services in Crandon, WI, has been approved for low level mercury testing. Three others are currently being evaluated. For more information about the mercury strategy or the emerging technology provision in NR 149, please contact Donalea Dinsmore by e-mail dinsmd@dnr.state.wi.us or by phone at (608) 266-8948.

Preservation requirements for volatile organics in wastewater samples

According to 40 CFR Part 136 and NR 219, table F - note 9, Wis. Adm. Code, volatile organic compounds (VOCs) must be analyzed within seven (7) days of collection if the sample is not acid preserved. The regulations imply that for a 14 day holding time, the purgeable aromatics and purgeable halocarbons need to be collected in separate vials and the aromatics must be preserved with acid to pH <2. In reality, laboratories that analyze halocarbons and aromatics concurrently are allowed to acid preserve the entire sample and apply the 14 day holding time for both aromatics and halocarbons. The distinction between the two is made because when halocarbons are analyzed as a separate fraction, acid preservation is unnecessary.

DNR initiates new round of permit applications

The Bureau of Watershed Management has begun mailing out another round of permit applications to many facilities. Laboratories that may be asked to analyze samples for the applications should have received a letter from the Bureau in November specifying the DNR's recommended methods for both priority pollutants and non-priority pollutants. Laboratories analyzing the non-priority pollutants listed in this letter by the recommended gas chromatography-mass spectrometry technique (SW-846 method 8270) are required to quantitate the samples using standards, just like the priority pollutants. Library matching for these compounds is not acceptable. Contact Donalea Dinsmore at (608) 266-8948 or by e-mail at dinsmd@dnr.state.wi.us for more information.





ANALYTICAL FOCUS: GROUNDWATER AND SOILS TESTING

Establishing baseline groundwater quality at landfills

The DNR's Waste Management Section in the Bureau of Remediation and Redevelopment has issued a clarification regarding the number of samples needed to establish baseline groundwater quality for volatile organic compounds (VOCs) at landfills. Section NR 507.18(3)(b), Wis. Adm. code states that if any well shows concentrations of VOCs greater than the limit of detection (LOD) in either of the first two quarterly sampling rounds, the well must be resampled for two additional times for a total of four sampling rounds. This applies to all VOCs with a few exceptions for "common laboratory contaminants". If a common laboratory contaminant is detected in both the sample and the method blank in either of the first two rounds, additional sampling is not required. In this case, the result must be qualified to indicate that the method blank is "out of control" per s. NR 149.14(3)(d). The following compounds are considered common laboratory contaminants:

- acetone
- methylene chloride
- methyl ethyl ketone (MEK)
- carbon disulfide
- phthalates
- methyl isobutyl ketone
- trimethyl benzenes
- naphthalene

The Bureau's experience shows that these compounds do not commonly occur as landfill contamination. Facilities must still sample an additional two times if any contamination other than the eight types listed above are detected. Facilities must also sample two additional times if any analyte, including those listed above, is detected in

the groundwater and also detected in a trip or field blank. For more information, please contact Janet Battista in the DNR's Bureau of Waste Management at (608) 267-3533.

Clarification of required holding times for diesel range organics

The Laboratory Certification Program is allowing the ten day solvent addition holding time for diesel range organics (DRO) at petroleum contaminated sites as previously noted in the Spring 1996 *LabNotes*. The current rule, NR 700.13, Wis. Adm. Code, states that solvent must be added to the samples within 72 hours of collection, but a published study confirms that the samples are stable for up to 10 days after collection. As a result, the Laboratory Certification Program is not enforcing the 72 hour holding time. The DNR's Bureau of Remediation and Redevelopment has proposed a rule change to NR 700 to correct the holding time, but the rule package is currently being reviewed by a technical advisory committee and will not be finalized for some time.

MICE from the EPA

The EPA's Methods Information Communication Exchange (MICE) is a service provided by EPA to answer questions on proposed and final methods in SW-846. The phone number is (703) 821-4690.





ANALYTICAL FOCUS: SAFE DRINKING WATER TESTING

Wisconsin updates drinking water rules

The DNR held public hearings in March regarding proposed revisions to ch. NR 809, Wis. Adm. Code pertaining to safe drinking water. Included in the proposal were revisions to tables A through H which contain approved test methods for drinking water samples and sample preservation requirements. The proposed changes mimic the EPA's final rule published in the December 5, 1994 Federal Register (Vol. 59 No. 232). Contact Mark Nelson of the DNR's Public Water Systems Section at (608) 267-4230 for a copy of the proposed changes.

EPA denies variance from nitrate preservation in drinking water requirements

The Department of Natural Resources' request to EPA Region V for a statewide variance from the nitrate-nitrogen sample preservation requirements has been denied. The State Laboratory of Hygiene (SLH) is in the process of obtaining additional information from states outside of Region V. If the information supports SLH's conclusion that there is no reason for preservation of drinking water samples, then the DNR will pursue a variance from EPA in Washington, DC. Currently, EPA Region V is requiring that laboratories either cool to 4° C and/or adjust the pH < 2 with sulfuric acid. The Laboratory Certification Program will continue to enforce this requirement for all laboratories unless a variance is granted.

How to reporting nitrate data in drinking water

Public drinking water facilities required to report nitrate data should report it as either nitrate-nitrogen or nitrate+nitrite-nitrogen.

These facilities must use the Department of Natural Resources' form 3300-232. The parameter code for nitrate-nitrogen is 618 and the parameter code for nitrate+nitrite-nitrogen is 630. Laboratories are required to fill out section III of the form. Please make sure that the name of the public water supply is on the form.



The Auditor's Corner

Alfredo Sotomayor, Senior Audit Chemist

Confirmation: Part II - The Quality of Mercy

Mercy is an unusual organic compound. It can be assayed by well-established protocols and can be detected by several gas chromatography (GC) detectors, gas chromatography-mass spectrometry (GC-MS), high performance liquid chromatography (HPLC)/ultraviolet detector, liquid chromatography-mass spectrometry (LC-MS), and infrared spectroscopy. Mercy is not commonly found in the atmosphere, is not a typical laboratory solvent, and is relatively stable, unlike its relative Percy, which becomes Perky when exposed to sunlight.

You analyze a water sample and find it has Mercy. Do you need to confirm the analyte in the sample? Consult this improvised oracle:

Truth Table: Do You Have Mercy, or Not?			
TYPE	Has this source shown any Mercy before?	Did you get Mercy's fingerprint?	Confirm Mercy?
I	YES	YES	NO
II	NO	NO	YES
III	YES	NO	NO/Your Call*
IV	NO	YES	NO/Resample or Reanalyze*
* Always subject to justification. The oracle cannot be held responsible for unexpected failures.			

The laboratory certification code (NR 149, Wis. Adm. Code) defines confirmation in the context of organic analysis and how definitive an analysis can be. It also gives an option of using knowledge about a sample source to decide whether confirmation is needed. But as you can see from the table, there are only two cases when the decision is categorical. When required, and this is important, the code dictates confirmation of the **presence** of an organic analyte. This means that we are compelled to verify the **identity**, not the magnitude of the compound. It is in this case, the quality of Mercy, not its quantity, that we seek to confirm.

I wish the world, and particularly, the world of environmental analyses, were simpler. Unfortunately, it is not. We and others use the term confirmation broadly to describe attempts at reproducing a quantity, or at verifying or dispelling a suspicion that an analyte detected in a sample comes from laboratory contamination or carry-over. The latter is the sense in which the term is used when re-analyzing soils preserved with methanol that have concentrations of volatile organic compounds between 25 and 60 ppb. Yet even for these samples, the established guidelines do not require reproducing a detected compound's concentration, beyond verifying that it is at or above 25 ppb.

Organic analyses that give fingerprints of compounds are more definitive than those that do not. A mass spectrum and an infrared spectrum are uniquely characteristic of an analyte, whereas a positive response by a flame ionization detector or an electron capture detector is not. So, analyses by mass spectrometry (MS) and any hyphenated technique using the MS detector, are more definitive. For other GC or HPLC detectors the more compound-specific the detector, the more definitive the analyses. When attempting confirmation by using different GC columns, the more dissimilar the stationary phase of the column, the more reliable the confirmation attempt.

How do you arrive at the correct decision in Type III and Type IV occurrences? You should decide by measuring the strength of your affirmatives and the weakness of your negatives. For Type III decisions, the reliability of your source information is paramount. Good site-history, one obtained or derived **independently** from the generation of analytical data, strengthens a finding and justifies avoiding the confirmation. For instance, knowing that Mercy is used by a specific client in an industrial process is independent information that could justify avoiding the confirmation in the same client's samples. When this information is not as categorical, then the specificity of the detector assumes more importance. For the most common detectors, my hierarchy from more specific to less specific is: PID, FLUORESCENCE, ELCD, ECD, NPD, FID, UV. You may have a different ranking, but your order should not be drastically different.

For Type IV decisions, where the identity of a compound is not questioned, in the absence of history about the sample, examining accompanying sample and quality control results is crucial. This can help rule out the possibility of a false positive resulting from laboratory or field contamination, or by carry-over. If quality control or other sample results suggest that the presence of a detected analyte is not legitimate, then resampling or reanalysis is the corrective action of choice, but neither of these are strictly confirmation, as defined in the code. Obtaining any information about the sample's history can moderate the need for reanalysis or resampling.

Once you have decided that confirmation is needed or desirable, then you must consider the sensitivity of the confirmation technique relative to the quantitative and reporting technique. But I will have mercy, and continue this discussion in my next column.

Secretary Meyer honors WP&L Edgewater, City of Medford

DNR Secretary George Meyer presented the Wisconsin Power & Light Edgewater Generating facility laboratory in Sheboygan and the City of Medford Wastewater Treatment Plant lab with the 1997 Registered Laboratory of the Year Awards at the March 26, 1997 Natural Resources Board meeting. According to Jack Sullivan, Chief of the DNR's Analytical and Statistical Services Section, the awards are presented annually to outstanding laboratories to recognize their commitment to producing high quality chemical data. This data is the foundation of many of the agency's decisions. This year's recipients demonstrated exceptional dedication to protecting Wisconsin's environment through their strong quality assurance programs and ability to use their data for improving day-to-day operations at their facilities. The Laboratory Certification and Registration Program would like to congratulate the award recipients and thank them for a job well done.

These laboratories were chosen from a strong pool of nominations this year, making selection of the recipients difficult. The Laboratory Certification Program would also like to thank everyone who nominated a facility and helped to make this year even more competitive than last! Nomination forms for the 1998 awards are available from the DNR's Central Office and are due prior to December 31, 1997. Contact Jeff Ripp at (608) 267-0579 or by e-mail at rippj@dnr.state.wi.us for more information about the awards or to request a nomination form.



DNR Secretary George Meyer (left) presents WP&L's Brian Gollhard (center) and Keith Deblaey (right) with the Lab of the Year Award for a larger facility.

Dean Woyak (center) and John Fales (right) of the City of Medford accept the Lab of the Year award for a smaller facility from Secretary Meyer.



Dealing with low level data: DNR answers questions about detection limits

Last January, section 149.15(3), Wis. Adm. Code, became effective. This section requires laboratories to quantitate and report data down to their limit of detection (LOD) for a list of specified analytes and specific programs (see Fall 1996 *LabNotes*). Since then, the DNR has fielded a number of very good questions about implementing this requirement in the lab. The Laboratory Certification Program feels that it is important to share the answers to these questions with all of the certified and registered labs in the program.

1) Do I have to report results down to the LOD for soils?

If you follow the five scenarios presented on page 7-1 in revision 5 (March 1996) of the Wisconsin Laboratory Certification Program: Program Information and Requirements manual (the "Yellow" book), logic follows that barring specific project or site requirements, a laboratory does NOT have to report results down to the limit of detection for soil samples. But like always, there are a few exceptions.

Soil samples from petroleum contaminated sites are one such exception. Section 9.4.4 of the *Wisconsin Gasoline Range Organics* (GRO) method requires laboratories to demonstrate that they can achieve a limit of detection of less than or equal to 25 µg/kg (wet weight basis) for methanol preserved volatile organics. For petroleum volatiles (PVOCs) in soil, laboratories are not required to report results below this level but they still must report their actual limit of detection. Similarly, section 10.2 of the Wisconsin GRO and section 9.2 of the *Wisconsin Diesel Range Organics* (DRO) methods require laboratories to demonstrate that they can achieve limits of quantitation (LOQ) below 10 mg/kg.

2) What do I do if my calculated LOD is unrealistic?

Many laboratories still appear to be uncomfortable with the language found in the note directly following section NR 149.15, Wis. Adm. Code. The note reads in part:

"...Laboratories shall use the best available analytical science to determine whether, in their best professional judgment, a substance has been detected."

The bottom line is that it's OK if the number achieved by performing the standard EPA Method Detection Limit (MDL) determination is a value that you believe is substantially lower (or higher) than what you, the chemist, truly believe is possible. But what do you do from there? The DNR is allowing laboratories to use their "best professional judgment" to determine what they feel their true limit of detection is. The only caveat is that if you use your judgment to adjust the calculated method detection limit, you need to be able to substantiate how you arrived at that decision.

As an example, suppose your calculated MDL for vinyl chloride is 0.004 ppb, but you **know** intuitively that if you were to prepare and analyze a standard at 0.004 ppb, your instrument would not even detect a peak. The next step in your logic then, is to determine what the instrument *can* detect. By analyzing standards prepared at 0.5, 0.25, and 0.1 ppb, you find that the first point at which an identifiable peak is obtained is at 0.5 ppb. You therefore use 0.5 ppb as your limit of detection.

(continued on page 13)

(Detection Limit, from page 12)

With metals determinations, particularly when using flame atomic absorption (AA), some other considerations are involved. The ratio of the 0.5 mg/L standard to the MDL (0.5/0.02) is 25. If we assume a linear response relationship, we can divide the response observed for the 0.5 mg/L standard (0.025) by this same factor and extrapolate to determine the expected response at the limit of detection. Under these assumptions, the expected response at the detection limit would be approximately 0.001 (0.025/25=0.001). Since this signal would not be distinguishable from a blank, this number is too low. We now look at the situation from

another perspective and theorize that a response of 0.003 to 0.004 would have to be obtained before we felt it was significantly different from a blank. Using the regression equation from the calibration data, an absorbance of 0.003 relates to a concentration of 0.070 mg/L, and an absorbance of 0.004 relates to a concentration of 0.093 mg/L. The only thing left to do is to analyze standards at these concentrations to verify the estimate.

For more information or assistance regarding limits of detection, contact Rick Mealy at (608) 264-6006 or by e-mail at mealr@dnr.state.wi.us.

DATA FROM FLAME AA (EXAMPLE)

MDL (40CFR Part 136, App. B)	=	0.02 mg/L
Response of 0.5 mg/L daily check standard	=	0.025 absorbance units
Response of 0.1 mg/L calibration standard	=	0.004 absorbance units
Response of typical calibration blank	=	0.000-0.001 absorbance units
Calibration responses (0, 0.1, 0.5, and 1.0 mg/L)	=	0.000, 0.004, 0.022, 0.043

Division of Health offers free OSHA consultation

Employers can sign up for free, on-site consultation to receive assistance in meeting current OSHA (Occupational Safety and Health Act) requirements. The main purpose of the consultation is to provide your employees with a safe and healthy work environment. As an employer, your only obligation is a commitment to take the necessary corrective actions in situations of imminent danger or conditions where a serious hazard is found. An imminent danger must be corrected immediately. In cases of serious hazards, the consultant and employer will develop an action plan for elimination of the hazard within a reasonable time schedule.

Consultation services are state and federally funded and offered through the Wisconsin Division of Health in conjunction with U.S. Department of Labor. To request these services, call (608) 266-0417. Contact Gordon Helmeid (608) 266-1818 for more information. Available consultation services include:

- Air sampling and analysis (to evaluate contaminants such as dusts, fumes, and vapors)
- Asbestos monitoring
- Blood-borne pathogens
- Surveying employee exposure (to hazardous substances)
- Emergency response and evacuation plan
- Evaluating the ventilation system
- Tuberculosis
- Hazardous energy (lockout/tagout)
- Information and training (in hazard communication, respiratory protection, hearing conservation, chemical hygiene plan, ergonomics, reproductive health, etc.)
- Noise monitoring
- Recommendations (for the control of occupational health hazards)
- Record keeping requirements
- Health and safety program assessment

Wisconsin auditors participate in interim NELAC meeting

The Second Interim Meeting of the National Environmental Laboratory Accreditation Conference (NELAC) took place last February in Bethesda, Maryland. During the interim meeting, the committees drafting the standards had a chance to work together, refine proposals, solicit comments and elicit reactions from participants. After the three days allotted to these discussions, the conference concluded with a fourth where the Environmental Laboratory Association Board, which gives direction to EPA and NELAC on related matters, held a separate meeting.

The conference was productive, and the participants continue to work diligently to have the standards ready for final approval during the third annual meeting, which will take place in Dallas on July 29 - 31. Standards on the mechanics of on-site assessments are essentially in final form. An assessors' training manual, reported to be halfway completed, should be available for review by June 15. The Quality Systems Committee, while still struggling with necessary minutiae, has drafted, reviewed, and revised a comprehensive set of standards. The Accrediting Authority Committee, which sets standards for the accrediting bodies, has mapped most of the process and intends to present some of the results as flowcharts. A member of the House of Representatives from the State of New Mexico is devising a survey for state legislators to solicit their ideas on implementation of the NELAC standards.

The NELAC Board of Directors expects that all standards will be sufficiently complete to become, if approved at the Annual Meeting, the basis for a national laboratory accreditation program. During the Annual Meeting in Dallas, representatives from the States, EPA, selected Federal agencies, and other regulatory programs will vote on the standards. It is speculated that at least four

states will be ready to implement the standards as a pilot program once they are approved. The State of Illinois is currently introducing legislation, modeled after the NELAC standards, to expand its current certification program beyond drinking water.

Continued topics of discussion in need of resolution before the Annual Meeting are: the definition of field of testing as the functional unit of accreditation; the granting and scope of reciprocity among accrediting bodies; the certification and oversight of multiple providers of proficiency testing (reference) samples; the determination of confidential business information during on-site assessments; the grandfathering of assessors (auditors) currently performing evaluations; the role of performance-based measurement systems (methods) in quality assurance and proficiency testing; the definition of viable sensitivity estimates (the detection limit issue is still hotly debated); and the composition and credentials of the team that will approve accrediting authorities, such as the Wisconsin Laboratory Certification Program, to manage local accreditation programs.

The State of Wisconsin will continue to participate in the national certification process and keep abreast of issues which may impact Wisconsin labs. We are enthusiastic about the progress made in setting standards but await more details on their implementation at the state level. Some aspects of the NELAC standards that would affect Wisconsin laboratories include:

- Requiring on-site evaluations every two years.
- Implementing rigorous data reporting requirements.
- Specifying detailed analytical and quality control requirements.

(continued on page 15)

(NELAC, from page 14)

- Increasing certification fees to handle additional administrative costs associated with reciprocity and a more frequent auditing schedule.

A national certification program is expected to reduce the number of audits a typical commercial laboratory would undergo because a single audit by the home state program will be sufficient to maintain recognition with other states. It is likely that fees will still be required from every state for each reciprocal certification a lab wishes to maintain. Assuming the standards are adopted in Dallas, Wisconsin would need approval from the Governor's Office and will likely have to propose a major rule change before it could adopt the NELAC standards. Regardless of the outcome in Dallas, the Certification Standards Review Council will be asked to take an active role in implementing national certification in Wisconsin.

The NELAC standards are available for downloading from the NELAC web site [<http://ttnwww.rtpnc.epa.gov/html/nelac/nelac.htm>]. We encourage Wisconsin laboratories to take some time to download and review the standards (they are in WordPerfect 6.1 format). We are very interested in receiving feedback from our registered and certified laboratories on this issue. Please drop us a line or contact your council representative to voice your questions or concerns. For more information about NELAC, contact either Rick Mealy (608) 264-6006 or by e-mail at mealyr@dnr.state.wi.us or Alfredo Sotomayor (608) 266-9257 or by e-mail at sotoma@dnr.state.wi.us.



DNR officially reorganizes to five regions

There are no buzzers, bells or flashing lights as the DNR implements some changes in the way its local offices will provide service to their communities. DNR Secretary George Meyer, however, says the agency has officially "flipped the switch" to a new organizational structure. According to Meyer, the department has adjusted the boundaries of its administrative regions to help better reflect the distribution of the state's natural communities and predominant land uses. Across the state, new regional boundaries are in effect, replacing six districts with five regions. Local government officials and legislators may notice some shift in the coverage areas for department offices. Large certified laboratories will not notice any changes as a result of the reorganization and they will continue to work directly with the Central Office in Madison. However, the change will affect many smaller certified and registered laboratories because they will be located in a new region and be assigned a new certification officer. Interested laboratories are urged to contact either their regional certification officer or Rick Mealy at (608) 264-6006 or by e-mail at mealyr@dnr.state.wi.us for more information.

World wide web sites

DNR Laboratory Certification

www.dnr.state.wi.us/eq/lc/

NELAC

ttnwww.rtpnc.epa.gov/html/nelac/nelac.htm

Code of Federal Regulations

www.nvi.net/CFRS/CFR/

US EPA Home Page

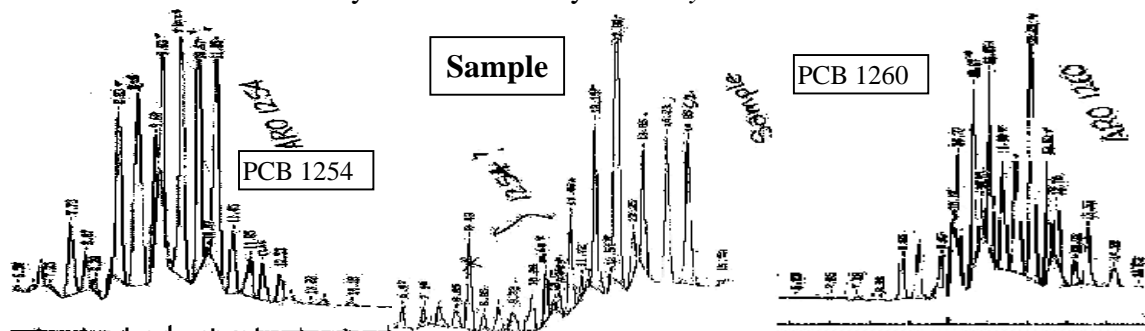
www.epa.gov/

Designer PCBs - when is a PCB not a PCB?

During a recent on-site evaluation, one of our auditors was shown a chromatogram that clearly appeared to be polychlorinated biphenyl (PCB) related, but did not match any of the standard chromatograms for the common PCBs or the nonroutine PCBs 1262 and 1268. The question posed to the auditor was, "Do we just report No Detect for each of the PCBs?". What a great question! The answer, of course, was not that easy. We showed the chromatogram to the most experienced PCB analysts at the State Laboratory of Hygiene, who offered some suggestions, including the possibility that what we were seeing was actually one or more polybrominated biphenyls (PBB).

While the laboratory is technically

justified in reporting "No Detect" for the PCBs, this is certainly a specific case that merits some sort of footnote to the analytical results. Perhaps in the future, samples of this nature would be best suited for congener analysis to determine if any of the known toxic congeners are present. Congener analysis is rapidly becoming state of the art as a replacement for the conventional technique of identifying and quantifying specific PCBs. We appreciate the type of information exchange that resulted from this on-site evaluation – it is from these experiences that we all learn more. For more information, contact Rick Mealy at (608) 264-6006 or by e-mail at mealyr@dnr.state.wi.us.



The middle chromatogram shows an example of a polybrominated biphenyl.

DNR releases new documents on common deficiencies, VOC monitoring

The Laboratory Certification Program has put together two new reference documents to help laboratories meet volatile organics monitoring requirements and to prepare for on-site evaluations. Both of these documents are available on the Laboratory Certification Program's web site or by calling Jeff Ripp at (608) 267-0579 or by e-mail at rippj@dnr.state.wi.us.

What VOCs do I need to monitor at my facility? Many of the DNR's regulatory programs require monitoring for volatile

organic compounds (VOCs). Since there are literally hundreds of different compounds that fall into this class, the Laboratory Certification Program has put together a list of VOCs commonly requested from the programs. Essentially, a laboratory could routinely monitor for these 68 compounds and cover all DNR programs except solid waste assessment monitoring, wastewater permit applications, and Appendix 9 of the federal hazardous waste code.

(continued on page 17)

(Documents, from page 16)

List of Common Deficiencies. This is a fictitious report of an on-site evaluation for a laboratory that doesn't exist. The Laboratory Certification Program has created this report by listing some of the most commonly observed deficiencies encountered during

laboratory audits. The report is intended to familiarize laboratories with the format and content of a typical audit report so they can understand the key elements scrutinized during an on-site evaluation. The report is not intended to be a substitute for reading and understanding Ch. NR 149, Wis. Adm. Code, or the approved methods of analysis.

NEWS BRIEFS

Correction!

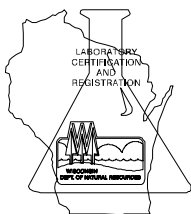
In previous editions (and on our web page) we inadvertently listed the wrong telephone number for Brenda Howald, the regional auditor in the former Southern District. Brenda's correct telephone number is (608) 275-3328. Our apologies to Brenda and any of you who have tried (unsuccessfully) to contact her.

Ordering EPA methods

Many of EPA's analytical methods are now available on diskette or CD-ROM instead of paper copies. Due to reductions in their printing budget, paper copies are only being supplied (for a fee) if specifically requested and copies are available. The EPA's Office of Water web site [<http://www.epa.gov/OW/>] contains information about how to order many other resources including trace metals methods, drinking water methods, pulp and paper industry methods, nonconventional pesticides methods, measurement of radioactivity in water, whole effluent toxicity methods, pharmaceutical industry methods and guidance on establishing trace metal clean rooms.

Revised application form available

The Laboratory Certification has printed new application forms to coincide with the newly approved fees for fiscal year 1998. Any laboratory submitting a new or revised application should check to make sure that the form they are using has the correct fees listed. Any application received after April should include the new fees. The correct application is dated as "revision 4/97" at the bottom right corner of the first page. Please discard any copies of the old forms. Application forms are available by contacting John Condron at (608) 267-2300.



LabNotes - Spring 1997

Wisconsin Department of Natural Resources
101 S. Webster St.
P.O. Box 7921
Madison, WI 53707

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